TRANSITION METAL DOPED TiO_2 NANOSTRUCTURES: SYNTHESIS AND STUDIES ON THEIR ELECTRICAL, OPTICAL AND DIELECTRIC PROPERTIES FOR SOLAR CELL APPLICATIONS

A

Synopsis

Submitted To

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2022

DECLARATION

I hereby declare that the work embodied in the synopsis entitled **"Transition Metal Doped TiO_2 Nanostructures: Synthesis and Studies on their Electrical, Optical and Dielectric Properties for Solar Cell Applications"** presented my contribution under the guidance of **Dr. Sudhakar Panday.** The matter presented in this synopsis has not been previously submitted for any other Degree/ Diploma to this or any other University.

Wherever prior works of others have been cited, it has been noted and listed in the bibliography.

I, hereby further affirm that all information in this document has been acquired and presented in accordance with academic standards and ethical conduct.

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1. Introduction

The process of "green synthesis" is an exciting technique in current years for synthesis of nanostructured materials. Metal oxide semiconductors viz. TiO_2, MgO, ZnO, CuO, CdO and NiO widely employed in recent years and are made through physical, chemical, and biological processes. One of them, Titanium Dioxide nanoparticles (TiO_2 NPs) is a well-known semiconductor with a broad bandgap about 3.2 eV for anatase and 3.0 eV for rutile phase [1], but the brookite phase is difficult to synthesize [2]. Although the crystal structure of TiO_2's anatase and rutile phases is tetragonal, that of the brookite phase is orthorhombic [3]. Due to its unusual characteristics such as its interconnecting pores and vast surface area, TiO_2 is frequently employed in cosmetics, photocatalysts, drugs, sensors, and solar cell applications [4]. The efficiency of solar cell depends on optical, electrical and dielectric properties of materials [5, 6]. Plants extract provides a better option for nanoparticles synthesis as they are free from toxic chemicals and contain natural capping agents [7]. The plant coriander (Coriandrum sativum L. is a member of the Apiaceae (Umbelliferae) family which is mostly grown from seeds all year long [8]. Coriandrum sativum leaves extract has several pharmacological effects such as anti-fertility, antidiabetic, antihyperlipidemic, antioxidant and hypotensive activities [9, 10]. Mentha spicata, also known as spear mint, is a tiny herbaceous perennial plant in the Lamiaceae family [11]. Indians frequently use it as herb because of its powerful antioxidant and antibacterial qualities [12, 13]. Trigonella foenum-graecum known as fenugreek is plant in the Fabaceae family with three tiny, obovate to oblong leaflets. It has antimicrobial and antioxidant properties [14]. According to phytochemical investigations, the main elements of the leaf extracts responsible for the decrease and stabilisation of nanoparticles were phenolic, flavonoid and terpenoid [15, 16]. The primary benefit of employing plants extract for nanoparticle synthesis is that this technique is bio-secure, simple to use, and contains a wide variety of metabolites that are engaged in the ion reduction process [17].

2. Statement of the Research Problem

There is a primary requirement to enhance the efficiency of solar cell devices as it is used to transforms sun energy into electricity which is widely applicable for utilization of renewable energy. Researchers did a lot of efforts to improve its

efficiency by various techniques, therefore it is very important to study for improvement of electrical, optical and dielectric properties of semiconductor oxides by doping of transition metals in the presence of surfactants and plants extract.

3. Literature Review

Bagheri et al. (2015) synthesized silver doped-titanium dioxide nanoparticles by green synthesis method which was carried out in aqueous gelatin solution. The results indicated pure Ag-TiO_2 and TiO_2 powders in anatase phase with an average crystallite size of 11 nm and 15 nm, respectively [18]. **Kumar et al. (2016)** synthesized Ag doped TiO_2 nanoparticles using rambutan peel extracts for their photocatalytic activity on methylene blue (MB) dye. TiO_2 and Ag doped TiO_2 NPs reported spherical in shape and size range of 100-180 nm and 30-200 nm, respectively. Ag doped TiO_2 NPs improved the photocatalytic degradation 81.6%, k=0.002495 min^{-1} and reported absorbance peaks in the range of 370–460 nm, which results in a reduction of the band gap energy and recombination rate [19]. **Jegadeeswaran et al. (2016)** synthesized Ag doped TiO_2 NPs using Padina tetrastromatica extract by green synthesis method. Ag doped TiO_2 nanoparticles were of anatase phase and average particles size reported 25±4 nm [20]. **Gopinath et al. (2016)** synthesized pure TiO_2, Au and Pt doped TiO_2 nanoparticles by green synthesis using Terminalia arjuna bark extract. The average crystallite size reported 7.18, 8.41 and 6.46 for pure TiO_2 Au and Pt doped TiO_2 NPs. The expected TiO_2 inter band absorption below 306 nm, Au doped TiO_2 shows surface plasma resonant (SPR) near 555 nm and platinum doped TiO_2 NPs shows absorbance peak at 460 nm [21]. **Alvaro et al. (2017)** synthesized Cu-doped TiO_2 nanoparticles, where TiO_2 nanoparticles by green synthesis method and Cu-TiO_2 nanoparticles were produced using a wet impregnation technique. SEM showed the spherical form with an average size between 27.3 and 70 nm. The band gap energy obtained for TiO_2 nanoparticles is about 2.98 eV and Cu doped TiO_2 with 1%, 3%, and 5% are 2.6 eV, 2.23 eV and 1.99 eV, respectively**,** Cu doped TiO_2 from 2.98 eV to 1.99 eV showed a reduction in the band gap, which shifted the absorption edge of TiO_2 toward a field of lower energy [22]. **Raorane et al. (2017)** synthesized Cu doped TiO_2 nanoparticles using fruit peel extract of Annona squamosa by green synthesis method that decreases the band gap up to ~2.17- 2.50 eV and shifts the absorption visible range of solar spectrum. The crystallite size was

obtained as 28.53 nm. [23]. **Hariharan et al. (2019)** synthesized Au and Pd doped TiO_2 nanostructures using aloe vera gel by green hydrothermal approach. The crystallite size is 47 nm for Au doped TiO_2 and 42 nm for Pd doped TiO_2 nanoparticles. Au doped TiO_2 reported band gap 2.29 eV resulted better performance compared to Pd doped TiO_2 reported band gap 2.58 eV [24]. **Rao et al. (2019)** synthesized Ag doped TiO_2 using aqueous extract of Acacia nilotica. TiO_2 and Ag doped TiO2 reported circular in shape, size of 9 nm and 17 nm, respectively. The band gap energy of Ag & Ti reported 3 eV and 4.5 eV, respectively [25]. **Solaiyammal et al. (2019)** synthesized Au doped TiO_2 by green synthesis route with the aqueous extract from the leaves of Phyllanthus embilica. Au doped TiO_2 are made of mixed anatase and rutile phases. Au doped TiO_2 nanocomposites was spherical in shape and particle size reported as 21.05 nm. Absorption maximum peak of pure TiO_2 and Au doped TiO_2 nanocomposite film at 368 nm and 536 nm, respectively [26]. **Mahboob et al. (2021)** synthesized gold (Au) and platinum (Pt) metal doped TiO_2 NPs using Enterolobium saman bark extract. All TiO_2, Au doped TiO_2, and Pt doped TiO_2 NPs were reported spherical morphology with an average particle size of 12, 10, and 8 nm, respectively [27]. **Nguyen et al. (2022)** synthesized Ag doped TiO_2 nanoparticles using an aqueous extract of Cleistocalyx operculatus leaves by green synthesis method. Ag nanoparticles reported particle sizes of 20-40 nm, which homogeneously enclosed the surface of the TiO_2 nanoparticles [28]. **Liminana et al. (2022)** synthesized Ag doped TiO_2 nanoparticles using Eucalyptus globulus L. extract through sol-gel and microwave-assisted sol-gel method. TiO_2 nanoparticles with sizes from 11 to 14 nm were obtained in the anatase phase. As compared with the pure TiO_2, there was no reduction of the bandgap for the Ag-TiO_2 was reported between 3.1–3.2 eV [29]. **Nethravathi et al. (2022)** synthesized TiO_2 and Ag doped TiO_2 nanoparticles using Cucumis melo (Muskmelon) juice by green combustion method. The average crystallite size of TiO_2 and Ag doped TiO_2 are 9.09 nm and 22.8 nm, respectively. The micrograph of Ag doped TiO_2 reported spherical shape with average particle is about 9.12 nm. TiO_2 and Ag doped TiO_2 nanoparticles reported absorbance peak at 458 nm with band gap 2.69 eV and weak absorbance peaks at 486 nm with band gap 2.53 eV and absorbance peak 700 nm with band gap 1.76 eV in the visible region, respectively [30].

4. Gaps in Research

From the literature survey, there is very few research work has been done on transition metal doped TiO_2 nanostructures by green synthesis method using anionic, cationic and nonionic surfactants and plants extract. There is no such detailed study of electrical, optical and dielectric properties of doped TiO_2 nanostructures reported so far under such synthesis condition.

5. Needs of the Study

The need of study on doped TiO_2 semiconductor nanostructures by green synthesis method for solar cell applications is required to obtain suitable band gap in the range of 2.10 - 3.00 eV, spherical morphology, larger surface area by reducing size of particles, and to obtain absorbance in range of 200-700 nm. Synthesis of doped TiO_2 nanostructures by using surfactant and plants extract will be very effective approach for enhancing the electrical, optical and dielectric properties in resultant to improve the efficiency of solar cell devices.

6. Objectives

1. To synthesis transition metal (Ag, Cu, Pt, Au) doped TiO_2 semiconductor nanostructures by green synthesis method using surfactants and plants extract.
2. To study morphological & crystallographic characteristics of the synthesized doped TiO_2 nanostructures.
3. To investigate electrical properties of the synthesized doped TiO_2 nanostructures.
4. To investigate optical and dielectric properties of the synthesized doped TiO_2 nanostructures.
5. To study effect of doping concentration on electrical, optical and dielectric properties of doped TiO_2 nanostructures.
6. To study effect of plants extracts on size and shape of doped TiO_2 nanostructures.

7. Scope

The scope of the work is to enrich the available knowledge on photovoltaic properties of doped TiO_2 using surfactant and plants extract. It has been reported in the literature survey, there is very less research on metal doped TiO_2 using surfactant and plants extracts. The work were based on the synthesis, characterization of various metal oxides like silver, platinum, gold and copper dopants with TiO_2 which explains the influence by its structure, phase, quality i.e. crystalline or amorphous nature,

morphology, particle size, surface area and band gap. The aim of this study is to develop superior quality of modified TiO_2 based nanostructures for high efficacy solar cell applications.

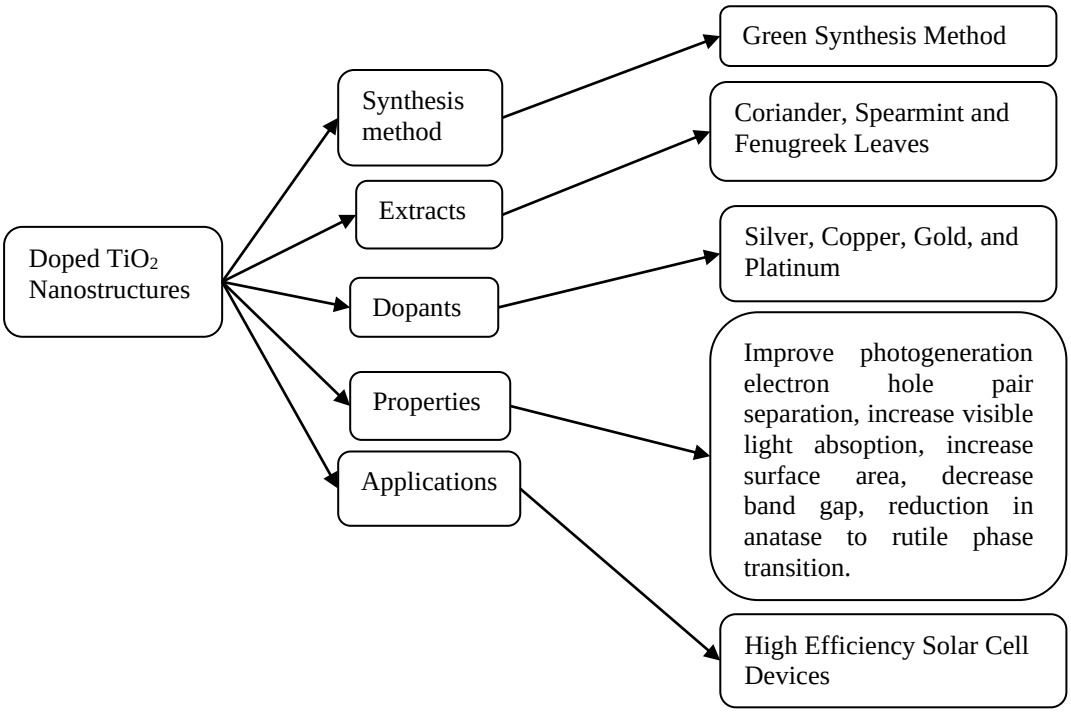
Doped TiO2 Nanostructures
Synthesis method
Extracts
Dopants
Properties
Applications
Green Synthesis Method
Coriander, Spearmint and Fenugreek Leaves
Silver, Copper, Gold, and Platinum
Improve photogeneration electron hole pair separation, increase visible light absoption, increase surface area, decrease band gap, reduction in anatase to rutile phase transition.
High Efficiency Solar Cell Devices

Figure 1: Synthesis methods, dopants, properties to be studied and applications of doped TiO_2 nanostructures.

8. Research Questions

- What is the structure of doped semiconductor nanostructures?
- What conditions affect shape and size of doped TiO_2 nanostructures?
- How are the results of the study going to help to get efficiency better than others in solar cell applications?
- How does the dopants and plants extract affect the electrical, dielectric and optical properties?

9. Research Design

(i) Research Methodology

Chemicals: Coriander Leaves (Coriandrum sativum L.), Spear mint Leaves (Mentha spicata), Fenugreek Leaves (Trigonella foenum-graecum),Titanium (IV) isopropoxide [$Ti(OCH(CH_3)_2]_4$, ethanol (CH_3CH_2OH), Copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$), Silver nitrate ($AgNO_3$), Hydrogen tetrachloroaurate(III) trihydrate

($HAuCl_4.3H_2O$), Potassium tetrachloroplatinate(II) (K_2PtCl_4), Cetyl trimethyl ammonium bromide (CTAB)/Sodium dodecyl sulfate (SDS)/Polyvinyl pyrrolidone (PVP), distilled water.

Preparation of doped TiO_2 Nanoparticles:

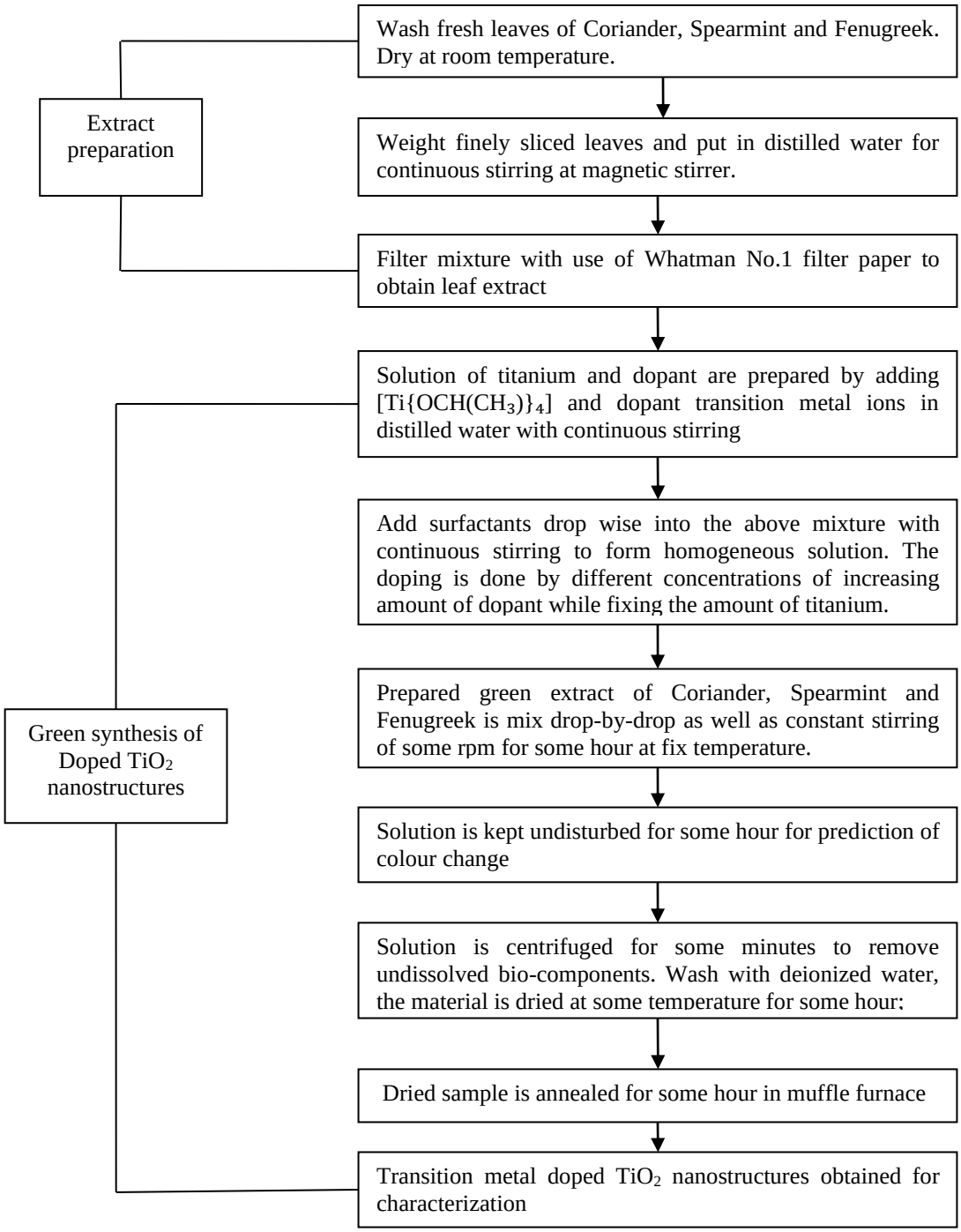
Wash fresh leaves of Coriander, Spearmint and Fenugreek. Dry at room temperature.
Extract preparation
Weight finely sliced leaves and put in distilled water for continuous stirring at magnetic stirrer.
Filter mixture with use of Whatman No.1 filter paper to obtain leaf extract
Solution of titanium and dopant are prepared by adding [Ti{OCH(CH3)}4] and dopant transition metal ions in distilled water with continuous stirring
Add surfactants drop wise into the above mixture with continuous stirring to form homogeneous solution. The doping is done by different concentrations of increasing amount of dopant while fixing the amount of titanium.
Green synthesis of Doped TiO2 nanostructures
Prepared green extract of Coriander, Spearmint and Fenugreek is mix drop-by-drop as well as constant stirring of some rpm for some hour at fix temperature.
Solution is kept undisturbed for some hour for prediction of colour change
Solution is centrifuged for some minutes to remove undissolved bio-components. Wash with deionized water, the material is dried at some temperature for some hour;
Dried sample is annealed for some hour in muffle furnace
Transition metal doped TiO2 nanostructures obtained for characterization

Figure 2: Steps for synthesis of doped TiO_2 nanostructures by green synthesis method.

(ii) Measurement and Scaling

The parameters like lattice parameters, crystal structure, morphology, optical absorbance, band gap, average crystalline size, average particle size, electrical conductivity dielectric constant etc. will be evaluated.

Structural Characterization

X-Ray Diffraction (XRD)

The lattice constant, XRD pattern, crystal structure and size of particles for the prepared samples of doped TiO_2 nanostructures will be studied.

Morphological and Compositional Analysis

- **Scanning Electron Microscopy (SEM)**

  Surface morphology of the prepared samples will be examined by SEM.

- **Energy Dispersive X-ray Analysis (EDX)**

  The compositional analysis of the prepared samples will be examined by EDX.

- **Transmission Electron Microscopy (TEM)**

  The shape, average particle size, Selected Area Electron Diffraction (SAED) pattern of the prepared samples will be studied by TEM.

Optical Properties

- **UV-Visible Spectrophotometer**

  Formation of the doped TiO_2 nanostructures will be characterized by calculating the energy band gap of the prepared samples from UV-Visible spectrophotometer.

Electrical Properties

- **Electrical Conductivity (EC) meter**

  D.C. Electrical conductivity will be carried out using EC meter in the suitable temperature range of the prepared samples.

Dielectric Properties

- **LCR impedance Analyzer**

  The dielectric constant and dielectric loss of the prepared samples will be measured using LCR impedance analyzer in the suitable frequency range.

(iii) Expected Outcomes

The expected outcomes are decreasing of band gap in range from 3.2 to 2.2 eV and absorbance band range between 200-600 nm in doped TiO_2 nanostructures. It can be beneficial for increasing efficiency from 14% to 20% by tuning optical,

electrical and dielectric properties of doped TiO_2 nanostructures.

(iv) Timelines

Activity / Months	**10 Nov. 2022 to 31 Mar.2023**	**1 Apr. 2023 to 31 Aug.2023**	**1 Sep. 2023 to 31 Jan.2024**	**1 Feb. 2024 to 30 Jun.2024**	**1 Jul. 2024 to 31 Nov.2024**
Preparation of samples					
Characterization of powder samples and analysis for various structural parameters.					
Measurement of optical, dielectric and electrical properties of samples and their analysis. Analysis of result and conclusions.					
Writing and publication of research papers					
Thesis writing					
Binding and submission of thesis					

(v) Tentative Chapter Plan

Sr. No.	Chapter
1	Introduction
2.	Literature review
3.	Experimental and characterizations techniques
4.	Results and discussion
5.	Conclusion and Future scope
6.	References

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